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OPERATIONS
SUPPORT

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PROGRAM NUMBER : 920102
PROGRAM NAME : Operations Support
PROGRAM COORD. : R. N. Ferguson
WRITTEN BY : Contributors
PERIOD COVERED : Fourth Quarter, 1992

Coordinator Summary: The longer term activities are progressing according to plan. Many short term requested studies are also being addressed. Due to both the diversity of areas covered and the numbers of individual projects, some study specific comments are included in this section.

- Important progress continues in the area of implementation of new procedures for obtaining Phytosanitary certificates.
- Research on cigarette beetle physiology is providing a better understanding of the interaction of methoprene with its target organism.
- Materials such as PG, propyl paraben, and sorbate appear to be responsible for the microbial resistance observed in wet tobacco materials.
- Microbial "hot spots" could not be confirmed in the process points of the NBL process which were examined.
- The hplc method for MH–30 may need further review to demonstrate to outside groups that it can be transferred to any laboratory. This issue is now being addressed in the crop protection agent (CPA) laboratory.
- The initial objective of the CPA laboratory will be completed as scheduled.
- Work with pesticide assay kits using the ELISA procedure was concluded this quarter.
- Continued support was provided to the Cooperative Leaf program for a large number of Bright tobacco varieties.
- Expertise gained as part of the adhesive specifications program has been applied to technical support issues such as stick–no–stick adhesive.
- Flavor specification negotiations with vendors are over 77% complete. Considerable interaction with the Flavor Center and other areas is ongoing.
- Customer complaints were addressed using a wide range of skills including analytical, entomology, and microbiology.
- Analytical methods necessary for unauthorized product studies have now been developed and the program is in place.
- Subjectives on the replication of the water transfer study on Marlboro cigarettes are complete and the data is being analyzed. Results will be available in early 1993.

AREA OBJECTIVE: Provide support to PM operations for technologies to insure leaf, blend, or reconstituted leaf quality. These include entomology studies, analysis for insect growth regulators or crop protection agents, and specialized analytical studies.

I. **Objective:** To provide entomological support in a timely manner to PM USA.

A. **Strategy:** Customer complaint (CC) evaluation from the Product Audit Facility.

1. **Results:** Twenty–five CC's were evaluated for insect infestation. About one–half of these were due to CBs while the other half came from other insects. One complaint was due to a carpet beetle infestation.

2. **Plans:** Continue to evaluate CC's as requested.

3. **Conclusions:** Appropriate documentation was sent to Product Audit personnel.

4. **Contributors:** S. Tenhet, D. Faustini.

5. **References:**

   Tenhet, S. W. Notebook No. 9091, pp. 72 and 73.

B. **Strategy:** Phytosanitary certification of export cut filler via conditioning rather than fumigation.

1. **Results:** Received final approval from the USDA/ARS/PPQ regarding acceptance for a new method of treatment of export cut filler using a 2–step conditioning cycle as a quarantine treatment for the tobaccos processed at Stockton St./Westab Facilities.

2. **Plans:** Monitor the implementation of this program.

3. **Conclusions:** As a result of this authorization, Philip Morris U.S.A. can now cease the use of methyl bromide fumigations and begin using the 2–step vacuum–steam conditioning cycle for export cut filler to obtain a Phytosanitary Certificate.

4. **Contributors:** M. Tickle and D. Faustini.

5. **References:**

   Faustini, D. L. and M. H. Tickle. Follow–up/Monitoring of the Phytosanitary Certification of Export Cut Filler Program at Stockton St.–Westab. Memo to C. K. Ellis; November 12, 1992 (in management review).

C. **Strategy:** Assist PM USA and PMI as requested.

1. **Results:** Completed a visit to Ecuador (TANASA, Tabacalera Andina, S.A.) to review current CB pesticide control practices.

2. **Plans:** Continue to support PM USA and PMI as requested.

3. **Conclusions:** A memo will be issued detailing the results and recommendations from this visit.

4. **Contributors:** D. Faustini

5. **References:**

   Faustini, D. L. Review of Pest Management Practices in Ecuador. Memo to H. Ganteaume; November 6, 1992 (in management review).

II. **Objective:** To develop a better understanding of the interaction of methoprene and/or other CB control agents on stored tobacco.

A. **Strategy:** Investigate the different physiological responses of some CB populations with respect to methoprene.

1. **Results:** (a) Esterase activity in the putative yeast CB symbiont *Cryptococcus albidus* was shown to be elevated in the presence of S–methoprene. (b) Feral CBs were collected from a "Kabat warehouse" and reared for several generations on methoprene–treated (3–80 ppm) and untreated, flue–cured tobaccos. F_3 generation adults were obtained from all the tobaccos and reared on untreated, flue–cured tobacco. The F4 generation adults were placed on methoprene–treated (11, 15 and 51 ppm) and untreated, flue–cured tobaccos. Adults (F_5 generation) were obtained from all tobaccos.

2. **Plans:** (a) No further work will be conducted with the yeast CB symbiont pending the outcome of the genetic studies with methoprene (no data reported). (b) The F_6 and F_7 generation first instar larvae will be placed on untreated, flue–cured tobacco (the second consecutive time these insects have been reared on non–methoprene–containing tobacco) and allowed to develop into adults. The F_8 generation, first instar larvae will be divided into two populations of insects. One–half will be reared on methoprene–treated (15, 51 and 80 ppm), flue–cured tobacco and the other half of the population will be reared on non–methoprene–treated, flue–cured tobacco. The data from this experiment will reveal the stability of the aberrant response of some CB populations to methoprene.

3. **Conclusions:** (a) The increased esterase activity of the yeast CB symbiont may provide the CB with the ability to feed on methoprene–treated tobaccos. (b) None at this time.

4. **Contributors:** S. Tenhet and D. Coar.

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5. **References**

Coar, D. Notebook No. 9112, p. 96.

III. **Objective:** At the request of Technical Services personnel, determine if wet tobacco materials (WTM) can be stored and sent to Park 500 instead of disposing of them.

A. **Strategy:** To evaluate the microbial changes, if any, during the storage of wet tobacco materials (>15% OV) obtained from the cutting area of Primary Processing.

1. **Results:** To date, no significant microbial changes have been observed from several storage studies in the laboratory and the field using a total blend WTM minus the ES, ET, and aftercut flavor. The WTM was held at ~23–25C with ~19–22% OV during all the storage studies. In addition to the microbial counts, selected chemical data (organic acids, IC sugars and TRS) also showed that no significant microbial development occurred during any of the storage studies.

To explain the lack of change in the microbial counts and chemical analyses, an experiment was performed which evaluated the preservative effects, if any, of the

WTM. A lyophilized extract of WTM was prepared, filter–sterilized, and challenged with a bacterial inoculum. No growth was observed from the mixture of undiluted extract plus inoculum. Some growth was observed from the mixture of diluted extract (1:6, extract:water) plus inoculum. However, even with the diluted extract the bacterial growth was not comparable to the bacterial counts observed from the control culture without extract. Since varying levels of known bacterial inhibitors such as propyl paraben, potassium sorbate, and PG are associated with the WTM, it was thought that these components were active in the lyophilized extract. The chemical analyses of the extract for these preservatives confirmed their presence in sufficient levels to inhibit bacterial growth.

2. **Plans:** A memo will be issued detailing the first set of laboratory and field storage studies. Additional samples of WTM are currently being held at 23C and 37.5C with 20%, 25% and 30% RH in order to determine if higher RH levels will facilitate microbial development.

3. **Conclusions:** The data from this series of experiments indicated that the preservatives on the WTM were preventing microbial development.

4. **Contributors:** J. Hutchison, D. Chadick , N. Thompson.

5. **References:**

Chadick, D. Notebook No. 9044, pp. 100–150.

IV. **Objective:** At the request of Reconstituted Tobacco Processing personnel, determine if any microbial "hot spots" existed in the NBL process.

A. **Strategy:** To microbially analyze samples obtained from different processing points in the NBL process in C Pilot Plant.

1. **Results:** Samples from the following process points were collected during sheet making runs before and after an extensive cleaning of the NBL pipes with citric acid:

1. aging tank #1 – outlet
2. aging tank #2 – outlet
3. aging tanks #1 and #2 – inlet
4. T3E24 into surge tank
5. ribbon blender

Along with the above–mentioned samples, portions of the dust, slurry, and finished sheet material were also obtained from the runs before and after cleaning.

The bacterial counts from the 5 process points were ~1–3 logs lower after cleaning compared to the samples collected before cleaning. Some mold was observed in the samples from the same process points before and after cleaning; however, the counts were in the range of 10–124 cfu/gm which are, by Tobacco Microbiology personnel standards, of little concern. No yeasts were observed from any of the process points before or after cleaning.

There were no significant differences in microbial counts from the dust, slurries, and/or sheets obtained before and after cleaning. Even though the cleaning

procedure did not eliminate all the microflora, certain members of the indigenous microbial populations were reduced by the cleaning efforts.

2. **Plans:** Finished sheet materials before and after cleaning will be made into cigarettes and subjectively evaluated.

3. **Conclusions:** From the data obtained (N=2 runs for most samples) it was not clear if microbial "hot spots" were found in the NBL process before cleaning. Cleaning the pipes used for the NBL process resulted in significant decreases in the microbial populations.

4. **Contributors:** D. Chadick, N. Thompson.

5. **References:**

   Chadick, D. Notebook No. 9044, pp. 100–150.

V. **Objective:** To provide methodology and measurements of crop protection agents (CPA) as needed to insure that tobacco product components and other materials meet regulatory requirements.

A. **Strategy:** Establish necessary existing and new methodology in the CPA laboratory.

1. **Results:** Four basic tobacco methods are now in place (FTR Part A, organochlorines, Part B, organochlorines and organophosphorus, and herbicides). All initial modifications are now complete and documented. The HPLC method for MH–30 has been implemented and has undergone preliminary evaluation. The FTR method for pendamethalin is in the process of undergoing laboratory evaluation.

   NCI techniques by gas chromatography–mass spectroscopy have shown considerable promise for confirmation of organochlorine CPA's in a sample matrix.

2. **Plans:** Perform TCLP methods as training exercise and review documentation in 1st quarter of 1993. Maintain basic FTR CPA capability. Complete review of HPLC MH–30 methodology by 12/31/1992. Continue the development of methodology for qualitative and quantitative confirmation of the FTR CPA's at regulatory limits by 12/31/93.

3. **Conclusions:** FTR methodology has been successfully integrated into the R&D environment.

   Confirmation of the FTR CPA's at regulatory limits continues to appear possible in samples matrices. Methodology for additional CPA's will await input from FTR.

4. **Contributors:** R. Davis, W. McCoy, G. Layman, J. Ware, N. Einolf.

B. **Strategy:** Provide documentation of methodology sufficient for laboratory operation and transfer where necessary.

1. **Results:** Draft methods for all FTR methods are complete in R&D ARD format and are awaiting review. TCLP methodology documentation will be maintained in

notebooks. FTR written method for pendamethalin is in hand for review. R&D HPLC method for MH–30 is in hand for review.

2. **Plans:** Complete documentation of revised FTR methods in R&D format by 12/01/92. Level of documentation for TCLP to be determined. Provide documentation of new methodology in R&D format within three months after commissioning. Time line is ongoing.

3. **Contributors:** R. Davis W. McCoy, W. Ryan, J. Periotti

C. **Strategy:** Provide technical support for other PM departments on CPA issues.

1. **Results:** Provided analysis for departments outside R&D for various samples. Performed purity analyses for QA–TQAF standards, assisting in occasional troubleshooting needs. Continue to maintain R&D, QA, FTR, and Leaf contacts regarding CPA issues as needed. TCLP methodology is being maintained as necessary for company needs.

2. **Plans:** Support for other PM departments is ongoing.

3. **Conclusions:** Support for QA–TQAF and other PM departments will be ongoing.

4. **Contributors:** R. Davis, W. McCoy, G. Layman, W. Ryan, J. Ware, C. McNeilly

VI. **Objective:** Provide results from reference methoprene hplc method as requested.

A. **Strategy:** Provide analytical data to Engineering to support studies monitoring application of Kabat to tobacco and tobacco materials.

1. **Results:** Methoprene and OV measured in samples from Rayco qualification test.

2. **Plans:** Continue analyses on samples submitted by engineering.

3. **Contributors:** L. Branch, T. Larus, M. Mangrum, R. McDaniel, W. Ryan

B. **Strategy:** Provide analytical data to entomological group (Project 1011) methoprene levels in tobacco samples.

1. **Results:** Methoprene analyses on tobacco samples from HHD cores from CB–infested Kabat warehouse 45; ELISA method development studies; and feeding media for CB bioassay studies.

2. **Plans:** Continue analyses on samples submitted by entomological group.

3. **Contributors:** L. Branch, T. Larus, M. Mangrum, R. McDaniel, W. Ryan

VII. **Objective:** Evaluate alternate technologies for trace component analysis on tobacco.

A. **Strategy:** Evaluate the Immunosystems EnviroGard™, 2–4D, Aldicarb, and Kabat Assay Kits

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1. **Results:** Aldicarb containing tobacco samples were re–tested to compare with the results obtained by FTR. The levels in the samples should have been 0, 0.2–0.4, 1.0–2.0, 2.0–3.0, and 5.0–7.0 ppm of Aldicarb, according to HPLC data obtained on these samples. The ELISA results indicated 0, 0.162, 1.089, 1.369 and 6.858 ppm Aldicarb respectively.

   Samples of oriental tobacco from warehouses 4 and 46 were also tested. Two samples were Kabat treated; the third contained no Kabat. These samples contained 5.14, 1.99, and 0.41 ppm respectively of Kabat as determined by ELISA.

2. **Plans:** Any additional work in the area of crop protecting agents will be conducted by PM Europe. Continue qualification of the Kabat assay.

3. **References:**

   Morrisette, E. C., PM Notebook 9176, pp. 183–190.

VIII. **Objective:** Evaluate Dianex levels in the plant environment.

A. **Strategy:** Utilize commercially available plate kit assay for Dianex assay.

1. **Results:** An extended preliminary study involved the collection of replicate tobacco dust samples at the same sample location initially selected for the preliminary study. Dust samples were collected at one month intervals to determine the stability and persistence of the Dianex® treatment two and three months after the initial application. The samples were analyzed for Dianex® concentrations with the EnviroGard™ Dianex® Plate Kit Assay. The results of the extended preliminary study suggest that the Dianex® treatment levels decrease with time. The majority of the samples contained Dianex® concentrations <3 ppm at the end of the three month test period. Dianex® levels <3 ppm are thought not to be efficacious for the control of the cigarette beetle. Based on the data collected from the preliminary study, it is recommended that the manufacturing plant environments be treated with a fresh application of Dianex® at no greater than one month intervals unless necessitated by high cigarette beetle trap numbers.

2. **References:**

   Gaines, O. Dianex® Analysis of Tobacco and Tobacco Dust Samples. Memo to D. T. Wagner, August 6, 1992.

   Gaines, O. Dianex® Analyses of Tobacco Dust Samples up to Three Months after Dianex® Application. Memo to D. T. Wagner, October 19, 1992.

   McCuen, R. Personal Communication to O. Gaines, October 16, 1992.

IX. **Objective:** To analyze samples submitted as part of the Cooperative Leaf program and provide data generated as a result of requested analyses.

A. **Results:** 124 samples were received from Clemson University as part of the South Carolina Insect Resistance Breed Line Test for the 1992 Bright Crop. These samples are presently being analyzed for Total Alkaloids, and Total Reducing Sugars by Continuous

Flow Methods and Total Nitrogen using the Model FP 228 Leco Nitrogen Determinator. The Bright Crop is normally received in September but this harvest did not come until October due to unusually heavy rains. 112 Bright tobacco samples were received from the Speight Seed Farms 1992 Breeding Lines of Winterville, North Carolina, for Alkaloids and Total Reducing Sugars using Continuous Flow analyses. 109 Bright tobacco samples were received from the R.G. Seed Company Oxford, North Carolina 1992 Breeding Line and are being analyzed for Total Alkaloids, and Total Reducing Sugars using Continuous Flow Methods.

B. Plans: Plans include continuing support as requested.

C. Contributors: C. Ament, T. Larus, L. Branch, R. Jones, M. Mangrum, K. Torrence, S. Langley, A. Ganzert, C. Callicutt

AREA OBJECTIVE: Provide technological support to PM Operations by the evaluation or specification of direct and indirect materials.

X. Objective: To determine the chemical compositions of materials used or proposed for use in cigarette manufacturing or found in our products.

A. Strategy: Perform appropriate infrared, X–ray, and other analyses in order to determine the chemical composition of the above materials.

1. Results: Numerous chemical identifications were made of materials used in the PM manufacturing facilities employing the appropriate analytical procedures. The results and the recommendations were entered into the Materials Evaluation database and reported to Quality Assurance. Materials analyzed included machine parts, cleaners, gaskets, and adhesives.

Cigarette package overwraps were examined by infrared procedures and identified as uncoated polypropylene or polyethylene terephthalate. The shiny "Flavor Seal" wrapper on RJR brands was identified as polyethylene terephthalate.

Numerous Marlboro packs and cartons were analyzed for project Fausto. Samples for this project were received from Hong Kong, Taiwan, Turkey, and the Peoples Republic of China. Trends and patterns have been evaluated and reported. The work will be continued.

2. Plans: The activity of the chemical component identification by instrumental methods in support of operation will be continued.

3. Contributors: S. Coleman, M. Griff, G. Vilcins

B. Strategy: Use light microscopy, scanning electron microscopy, and energy dispersive spectroscopy to analyze appropriate samples.

1. Results: Several types of insulating materials were identified and characterized. Other materials examined included powder collected from the surface of burlap–wrapped tobacco bales. The powder contained two types of particles. Most of the particles were cream–colored and contained Mg and Ca, but one contained P in

addition to the Mg and Ca. A second particle type was a black particle covered with a white powder. The black and white particles contained Si, Fe, Ca, Mg, and some Al.

2. **Contributors:** V. Baliga, D. Miser

3. **References:**

Baliga, V., PM. Notebook 8911, p. 92.

Baliga, V., Miser, D., "Characterization of White Powder on Burlap Bag," Memo to D. Watson, October 27, 1992.

XI. **Objective:** To provide technological support to PM USA for quality improvement by the evaluation or specification of direct and indirect materials. Provide specialized problem solving support.

A. **Strategy:** Identify the chemical composition of direct materials used in cigarette manufacturing employing several different analytical techniques.

1. **Results:** The analytical work to determine the chemical composition of adhesives used at PM facilities was continued. Problems with the stick–no–stick adhesives on the top flap of cartons were submitted for analyses by infrared and other procedures. Fiber tear appeared on both the top flap and the inside small flap. Minor differences in the CH–stretching region were seen in the board samples. The adhesives tended to separate into layers on standing. The top layer contained water, a starch, and possibly a polyamide among other materials. This layer was different from the total material, which was mostly polyvinyl acetate and other components. More work will be done.

   The program used to determine the lot–to–lot variability of the adhesives was is being evaluated by PM statisticians. Their recommendations should be available shortly.

   HPLC analyses were made for melamine, glyoxal, and preservatives in RL and RCB sheet materials for German specifications. The specifications group and Park 500 labs conducted a collaborative study to assure the results were consistent between the groups.

2. **Plans:** The program to analyze the adhesives and their lot–to–lot variability will be continued. Other analyses will be performed as necessary.

3. **Contributors:** B. Baronian, K. Dudzinski, M. Griff, C. Keene, G. Vilcins.

XII. **Objective:** Develop analytical and sensory specifications for incoming flavors used by PM USA. Transfer specifications and methodology to the Flavor Center and Technical Services.

A. **Strategy:** Work with Technical Services, Purchasing, QA, and other R&D staff as required to transfer specifications and methods as vendor agreements are obtained. Discuss specs and methods with vendors to reach agreements on same.

1. **Results:** Negotiations continued with the vendors. There are 300 DM codes which required written flavor specifications. Currently over 77% of DM code negotiations

are complete with the remaining DM codes under negotiation. Implementation of some of these specifications has begun at the Flavor Center. The specification group is working with the Flavor Center to adjust methodology where necessary and assist in the implementation as needed.

Biweekly meetings are attended by the specifications group at the Flavor Center with personnel from technical services, purchasing, regulatory and the Flavor Center. These meetings are intended to resolve any issues that may evolve and to help better organize the remaining specification process.

2. **Plans:** Continue to support Purchasing in obtaining agreements with vendors. Develop additional flavor specs as required.

3. **Contributors:** B. Baronian, K. Dudzinski, N. Einolf, K. Sanders, G. Vilcins

XIII. Objective: To determine the chemical composition of printing inks used on PM products in order to improve the subjective characteristics of the final product.

A. **Strategy:** Perform analyses on printing inks employing the purge–and–trap GC–MS techniques and any other analytical procedure in order to determine the composition of the components which could cause poor subjective results in the final product.

1. **Results:** A program was initiated by Purchasing Technical Services with the involvement of R&D Analytical Research Division, Flavor Technology Division, and other R&D representatives to develop a commercially viable offset printing system that will deliver subjectively acceptable materials with a high degree of confidence. The program is underway and proofs of inks and coatings on paper have arrived for analytical and subjective testing.

2. **Plans:** The P&T GC–MS procedures as well as other appropriate methods will be employed to analyze the samples from the offset printings.

3. **Contributors:** K. Dudzinski, C. Keene, G. Vilcins

XIV. Objective: To characterize packaging materials according to their morphology and elemental content

A. **Results:** Competitive brand flavor seal overwraps were evaluated and compared. The major difference between Vantage (white pack) and Winston (red pack) flavor seal was that Vantage contained one less layer of polymer on the inside surface. It also appeared that the Vantage flavor seal had twice as many holes in the Al layer. These holes, however, were covered and sealed by the polymer layers for both the Vantage and Winston overwraps.

B. **Contributors:** V. Baliga

C. **References:**

Baliga, V., "Vantage Metallized Film Overwrap Compared to Winston Regular Metallized Film Overwrap," Memo to P. Grantham, September 18, 1992.

AREA OBJECTIVE: Provide technological support to PM Operations for finished product quality. Investigate customer complaints as requested by Product Audit Facility.

XV. Objective: To determine if foreign materials are present and to characterize morphology and elemental content of the same in customer complaint samples.

A. Strategy: Use light microscopy, scanning electron microscopy, and energy dispersive spectroscopy to analyze the samples.

1. Results: Five customer complaint samples were examined. Three samples involved foreign materials in the cigarettes, one had a moldy smell, and one had a foreign substance on the carton. They included the following:

- A piece of wood in the shape of a tooth pick was found in one cigarette of Marlboro Lights KS FTB. No heavy elements were found on the wood which suggested that it was not an explosive load.
- Match heads were found in one broken cigarette of Genco Ultralight 100 SP. Nothing else was found in the two remaining intact cigarettes.
- Wires were found in Alpine Lights cigarettes. They were identified as electronic wires.
- "Moldy" cigarettes that were returned from China were examined. One cigarette from each of two packs contained vegetative mold growth. Cigarettes from two additional packs did not contain evidence of mold growth.
- A material that was identified as 'water glass' was found on two items, a. a piece of cardboard box and b. a carton of Virginia Slims. 'Water glass' is used sometimes as a box sealer.

2. Contributors: L. Thompson, V. Baliga

3. References:

Baliga, V., "Customer Complaint #92040," Memo to S.S. Yang, August 25, 1992.

Thompson, L., "Customer Complaint #92047," Memo to S.S. Yang, October 5, 1992.

Baliga, V., "Characterization of Customer Complaint #92052," Memo to S.S. Yang, November 11, 1992.

Baliga, V., "Customer Complaint #92046," Memo to S.S. Yang, October 23, 1992.

Thompson, L., Griff, M., "Customer Complaint #92054," November 5, 1992.

XVI. Objective: To characterize unauthorized cigarette products that were sold as US export. The project is a joint effort between IOS, Richmond R&D, FTR–R&D, and the sales force with the objective of identifying the number of and the source of unauthorized operations.

A. Strategy: Characteristics of each of the unauthorized and authorized products have been defined and the analyses have been divided between R&D–Richmond and R&D–Neuchatel by mutual agreement. Further actions have been defined and await approval.

1. **Results:** Twenty–nine unauthorized products have been received. Twenty–one of these were used to define the most useful characteristics to be used as a reduced set of analyses for future samples. Items examined within Analytical Microscopy included cigarette rods, tipping paper inks, tear tape pigments, blank inks, closure/stamp pigments, foil compositions and microstructure, tipping paper patterns, filters, mode of cigarette construction, method of pack construction, printing method, Marlboro logo patterns, tear tape type and composition, and tear tape cutter defect patterns. Information collected from other areas in R&D included packaging adhesives, tear tape adhesives, stamp adhesives, tipping adhesives, sideseam adhesives, humectants, flavors, beta–methylvaleric acid, nicotine, and triacetin. Fiber analyses of packaging materials and papers were solicited and received from an outside laboratory.

   All of these pieces of information have proved valuable in identifying the number of counterfeit operations responsible for the samples. In addition, the number of cigarette machines involved in the manufacture of the first 20 counterfeit samples was determined as well as the number of packing machines involved.

   This information was presented by Dr. D. Miser at the meeting of the Unauthorized Product Technical Committee in Neuchatel.

2. **Plans:** Further work will involve the examination of more unauthorized products and known vendors' products. This new information will be compared to the data base of information on the previously examined unauthorized products.

3. **Contributors:** D. Miser, V. Baliga, L. Thompson

4. **References:**

   Morgan, G.H., International Operation Services, "Meeting of Unauthorized Product Technical Committee," Memo to Distribution, October 27, 1992.

XVII. Objective: To characterize and identify component parts of competitors products.

A. **Results:** Smoking articles that were marketed as medicinal aids in Japan were examined and characterized. Two packs, NEO CEDAR Regular and NEO CEDAR KS, were examined and found to resemble typical cigarette construction. The shredded filler material, however, consisted of plant pith, soft wood fibers, monocot leaf pieces, and other, possibly dicot, leaf materials. Tobacco is a dicot leaf.

B. **Contributors:** V. Baliga

C. **References:**

   Baliga, V., "NEO CEDAR Smoking Articles," Memo to D. Watson, October 6, 1992.

XVIII. Objective: Determine if a relationship exists between a perception of 'stale' and the water content of the cigarette.

A. **Strategy:** Develop and apply procedures for the selective removal of water relative to the modification of natural and added tobacco flavors and correlate the extent of the changes with perceptions of 'stale'.

1. **Results:** Data produced during the initial study suggested that there is a quantifiable difference in sensory response between cigarettes that are at the same moisture content but which have achieved that moisture level via different routes.

   The sample preparation/evaluation protocol was duplicated to test the validity of the initial conclusions. The samples were prepared as in the first test, sensory evaluations have now been completed, and the results will be available for correlation to analytical data by the first part of 1993.

2. **Contributors:** R. Esperdy, M. Jeltema, F. Scott

NEW
EXPANDED TOBACCO

PROGRAM NUMBER : 920104
PROGRAM NAME : New Expanded Tobacco
PROGRAM COORD. : E. B. Fischer
PERIOD COVERED : Fourth Quarter, 1992

Coordinator Summary:

Batch NET – Impregnation and reordering Process Development work is complete. Expansion equipment development is behind the original schedule by two to three months. It is expected that results will be available for testing at Bermuda Hundred during start–up.

Parity at a 15% inclusion level of NET in Merit and Benson & Hedges compared to 12% DIET inclusion has been demonstrated this quarter in POL results. Additional full margin brand blend work is in progress.

An active program with European Leaf personnel started this quarter and will continue to support the BOZ Plant start–up in 1993.

A pilot test with Australian tobacco was conducted to investigate the feasibility of the first DIET plant retrofit. Support of the Australian effort will continue as required.

Continuous NET – Early delays in developing a mathematical model for the SCI Process have not been overcome. Results from the gaseous impregnation model, developed in the second and third quarters, proved helpful in gelling PM's position on CO_2 expansion processes and in resolving patent validity issues with the Japanese. These and other developments during the fourth quarter have helped to direct the 1993 program objective to optimally design and build a small scale, skid–mounted NET SCI process for International locations. This will require extended modeling work to simulate the entire, dynamic CO_2 process.

I. **Objective:** Define the Batch Gaseous Impregnation Process for Burley and Oriental.

A. **Strategy:** Define the batch gas process parameters for Burley.

1. **Results:** During this past quarter, the last four impregnation and expansion tests were made using 12% and 15% OV C34 Burley at impregnation pressures of 500 psig and 800 psig to determine the effect of impregnation pressure on equilibrium CV/OV. Analysis of these expansion results indicate that 500 psig impregnation pressure requires longer cycle times than 800 psig impregnation pressure.

2. **Conclusions:** The batch gas impregnation parameters for Burley are not significantly different than those for Bright.

3. **Contributors:** C. Moogalian, J. Dobbs, R. Lum, J. Atkinson, V. Covington, Materials Evaluation Lab, Semiworks Primary, G. Romig

4. **Plans:** Complete. No further work is planned.

B. **Strategy:** Define the batch gas process parameters for Oriental.

1. **Results:** Similarly during this past quarter, the last four impregnation and expansion tests were made using 12% and 15% OV MT Oriental at impregnation pressures of

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500 psig and 800 psig to determine the effect of impregnation pressure on equilibrium CV/OV. Analysis of these expansion results indicate that 500 psig impregnation pressure requires longer cycle times than 800 psig impregnation pressure.

2. **Plans:** Complete. No further work is planned.

3. **Conclusions:** Similarly, the batch gas impregnation parameters for Oriental are not significantly different than those for Bright.

4. **Contributors:** C. Moogalian, J. Dobbs, R. Lum, J. Atkinson, V. Covington, Materials Evaluation Lab, Semiworks Primary, G. Romig

II. **Objective:** Optimize the Humid Air Reordering and Precooling Processes for Commercial Scale–Up.

A. **Strategy:** Complete the pilot plant studies on humid air reordering and precooling.

1. **Results:** One process patent and two equipment patents were filed for the humid air reordering process.

2. **Plans:** No further work is planned.

3. **Conclusions:** Pilot plant process studies are complete.

4. **Contributors:** R. Lum, M. Toerne, Materials Evaluation Lab, Patent Department, W. Winterson

III. **Objective:** Determine a Tower System Design which will Ensure Successful Scale–Up of the NET Process.

A. **Strategy:** Develop a tower feed valve and separator which will provide maximum product expansion, uniformity, and subjective acceptability.

1. **Results:** The existing and new plant slice tangential separators were evaluated at high throughputs. The plant slice tangential provided plug flow separation at all throughputs. Work on the short cyclone was discontinued due to higher product breakage when compared to either tangential.

   The present close coupled feed valve has been tested against a standard feed valve. Results are pending.

2. **Plans:** Receive plan and elevation drawings for Bermuda hot end installation from Engineering by November 1992. Determine pilot plant design for a modified separator that will scale–up for Bermuda by December 1992. Construct and test a modified separator during December 1992.

   Transfer technology to PM Engineering by February 1993.

3. **Conclusions:** Although this work will be completed 3 months behind schedule, it will provide adequate time for testing at Bermuda during start–up.

4. **Contributors:** A. Kumar, L. Lipscomb, R. Lum, J. Washington, W. Winterson

B. **Strategy:** Determine the tower design and operating ranges which will ensure successful scale–up of the NET process.

1. **Results:** The 8" tower feed system was modified to increase tobacco throughput to cross–sectional loadings used in commercial DIET plants. Tests demonstrated a decline in product CV with increased throughput at equal exit OV. These tests also verified earlier results showing increased breakage with gas velocity. Velocity did not affect CV in these tests.

   A test grid was run at the Cabarrus DIET Plant to check these results on a 24" commercial tower. This work confirmed the drop in CV with throughput. A slight increase in breakage with increasing gas velocity was measured, less than that measured in the pilot plant. An unexpected result at Cabarrus was an increase in CV with gas velocity at 5000 lbs/hr throughput. This effect was traced to improved gas/tobacco contact in the first section of the tower.

   Work has begun on a pilot cornerless, rectangular tower in an attempt to improve gas/tobacco contact and provide improved CV at high throughputs.

2. **Plans:** Install a cornerless, rectangular tower during December 1992. Test and evaluate results from the rectangular tower by February 1993.

3. **Conclusions:** Although this work will be completed 2 months behind schedule, it will provide adequate time for testing at Bermuda during start–up.

4. **Contributors:** S. Barton, A. Kumar, R. Lum, R. Moffitt, E. Moss, W. Winterson

IV. **Objective:** Implement the Gaseous Batch Process Commercially.

A. **Strategy:** Assist Engineering in the implementation of the Batch NET process.

1. **Results:** Continued interaction and support were provided to PM Engineering for the BOZ and Bermuda installations.

2. **Plans:** Continue to interact with Engineering and Operating personnel as plant design continues.

3. **Conclusions:** R&D, Engineering, and Manufacturing personnel continue to operate together as a team with areas that need attention being addressed.

4. **Contributors:** P. Barton, J. Dobbs, J. Tilly, B. Forkins, R. Lum, D. McDowell, M. North, M. Toerne, W. Winterson

B. **Strategy:** Assist Leaf and R&D Flavor and Product Development personnel in developing use strategies for NET.

1. **Results:** The work of the first three quarters culminated in a successful POL for 15% inclusion of NET in both Merit and Benson & Hedges. Process Development continues to support Leaf, Flavor Technology, and Cigarette Technology to develop

use strategies for NET in the following brands: Virginia Slims, Virginia Slims Lights, Virginia Slims Ultra Lights, Saratoga, Parliament, Marlboro Ultra Lights, and Benson & Hedges Ultra Lights.

A series of 10 impregnation and expansion tests were made using No. 10 Bright for European subjective evaluation tests during November.

The first work has begun on the conversion of existing DIET plants to NET. During this quarter, 21 low pressure impregnation and expansion tests were made using uncased Australian DIET feed tobacco. Expansion temperatures were 425°F, 580°F, and 625°F. Approximately 4000 lbs of tobacco was shipped back to Australia. Cigarettes and subjective evaluation tests are scheduled for January 1993.

2. **Plans:** Continue expansion of blends of Bright, Burley, and Oriental as per request of Leaf and Product Development Departments. Additional expansion tests for the European program are expected during January 1993.

   Pending a successful outcome of the Australian pilot test, a preliminary low pressure NET factory test is planned in Australia before the end of the second quarter of 1993.

3. **Conclusions:** NET utilization strategies continue to be directed by New Product Development with the Leaf Group developing the blends, Flavor Technology and Domestic Product Development group the products, and Process Development providing the NET products and cigarette fabrication as requested.

4. **Contributors:** C. Moogalian, J. Dobbs, D. Leister, B. Riggan, B. Peace, B. Taylor, T. Clarke, V. Covington, J. Atkinson, P. Grantham, G. Carter, W. Mokarry, G. Romig, G. Inge, Analytical Research, Materials Evaluation Lab, Semiworks Primary and Make Pack Personnel, Chemical Research, Flavor Technology, R. Southwick

V. **Objective:** Develop the Short Cycle Impregnation process to produce an expanded tobacco material having equivalent or better physical and subjective attributes as compared to the Batch process.

A. **Strategy:** Develop the Short Cycle Impregnation process to provide uniformly impregnated product.

1. **Results:** During this past quarter, 36 impregnation tests were done using 12%, 15%, 16.5%, 18%, and 21% OV uncased Bright tobacco at pressures of 400 psig, 600 psig, 700 psig, and 850 psig to determine the optimum pressure and OV to run the SCI process. Based on the results of this current series of tests, the impregnation conditions are similar to the Batch NET process at 15% OV and CO_2 impregnation pressure of 750 psig to 800 psig.

   Similar to the impregnator model discussed last quarter, the entire SCI process is dynamic. It is obvious that a simulation of the entire process is necessary in order to optimize the process design. During this past quarter, considerable time and resources have been expended to determine a suitable simulation package.

   Results of the impregnator dynamic model were reviewed with International and Legal personnel to help formulate a resolution to gas patent validity issues in Japan.

2. **Plans:** The impregnator model will be converted from a single gaseous phase CO_2 model to a two phase model which includes the condensation and evaporation of liquid CO_2 within the impregnator. It is anticipated that the selection of process simulation software will be completed and implementation started in November. It is expected that a complete CO_2 process model will be available by April 1993.

3. **Conclusions:** The program has been redirected and scheduled for 1993 with the objective of optimally designing a small scale, skid–mounted expansion plant for International applications.

4. **Contributors:** D. Leister, J. Dobbs, K. Cho, W. Nichols, R. Prasad

B. **Strategy:** Evaluate process and product performance of the SCI Pilot Plant.

1. **Results:** No activity.

2. **Plans:** The plans continue to be to redirect the development efforts toward design of a commercial prototype.

C. **Strategy:** Evaluate scale–up feasibility on a commercial size prototype.

1. **Results:** The model development of the impregnator has led to an understanding of its operating characteristics and has provided a good scale–up tool. Due to patent concerns of the SCI process, the direction of scale–up is toward the NET process with higher tobacco packing densities. The selection of a properly designed impregnator to meet this objective and the simulation of the entire CO_2 process will be done in parallel.

2. **Plans:** The plans for the SCI process have been redirected to have an impregnator design by the end of the first quarter of 1993.

3. **Conclusions:** The design and construction of a prototype facility has been scheduled for completion by the first quarter of 1994.

PAPER
TECHNOLOGY

PROGRAM NUMBER : 920106
PROGRAM NAME : Paper Technology
PROGRAM COORD. : S. Baldwin
WRITTEN BY : S. Baldwin and G. Bokelman
PERIOD COVERED : Fourth Quarter, 1992

Coordinator Summary:

Wood Pulp Paper

Pyrolysis, thermal chromatography and pyrolysis–GC–MS analyses of several wood and flax samples were performed. No qualitative differences were found between the major products for wood and flax. However, some quantitative differences were observed for some sulfur–containing compounds. The wood pulp pyrolyzate generally yielded greater quantities of those components. Additional extensive investigations are planned to explore possible benefits for the development of improved wood pulp papers.

Cigarette Paper Specifications

Mathematical models relating FTC tar and puff count to cigarette paper parameters were used to design four papers which could replace the existing seven papers while maintaining 0.5mg FTC tar control increments. The papers will be made in mill trials at Kimberly–Clark in December.

An evaluation of machine and product performance using increased chalk (30% calcium carbonate) papers was conducted in the Manufacturing Center on high speed equipment. The 30% chalk papers ran as well at high speeds as the 26% chalk paper. Rates for paper related defects for the 30% chalk paper were not significantly different than those of the 26% chalk papers.

The machinability of papers with extreme tensile properties was investigated by running cigarette papers at varying tensile strengths on a Protos maker at 10,000 cigarettes per minute. Papers with tensile values of 0.07kg/mm or higher ran well at high machine speed; below that tensile level, the paper ran inconsistently or not at all.

Reduced Sidestream

Calcium Carbonate Papers

Sidestream visibility reductions of 67% to 72% were achieved for 23.0 and 24.0mm cigarette models using low permeability papers and/or lowered pH phosphate solutions to impregnate the papers.

Process conditions have been defined for the production of low sidestream papers at Kimberly–Clark's Spotswood mill. These conditions will be referenced in the specification for the 10–064A low sidestream paper.

A matrix study designed to quantify the effect of paper parameters on sidestream reduction and static burn time for 23.0mm cigarettes was completed. Calendering low sidestream papers was also shown to boost sidestream reduction.

No benefit was observed for sidestream reduction when ground calcium carbonate is used in low sidestream papers. No further work in this area is planned.

Magnesium Carbonate Papers

Sufficient aqueous non–sol–gel material was synthesized in the Chemical Research Division to permit the production of 3,000 meters of paper at the University of Maine. The papers have been slit into four inch rolls for coating. After coating the test and control papers, cigarette samples will be prepared.

I. **Objective:** Evaluate the feasibility of replacing flax papers with wood pulp papers for full margin brands and develop the appropriate papers.

A. **Strategy:** Conduct analytical and subjective evaluations of wood and flax pulps and papers.

1. **Results and Conclusions:** Descriptive panel evaluations were conducted for Marlboro KS prototypes made with four experimental wood pulp papers. The test papers included two Ecusta 50/50 hardwood/softwood papers with different hardwood pulps, and two Kimberly–Clark papers with 100% hardwood and 100% softwood fiber contents. All four wood paper models received negative attribute ratings (bitter, astringent, mouth coating) compared to the flax control model. Papers consisting of blends of flax and wood pulps were received for evaluation.

   Pyrolysis and thermal chromatography analyses were conducted for several wood and flax samples using GC–MS for product identification. No qualitative differences were found between the major products of wood and flax papers. However, quantitative differences were observed for some sulfur containing compounds, with the wood pulp paper pyrolyzate yielding the greater quantity of these compounds. Pyrolysis experiments were also conducted to develop baseline product patterns for three pulp components: xylan, mannan, and lignin. The lignin pyrolysis revealed a peak for SO_2 that is present for the wood paper but not for the flax paper. Based on these findings, analyses of mainstream smoke for sulfur–containing compounds is being conducted for models made with wood and flax papers. Levels of sulfur, chlorine, and nitrogen in papers and pulps are being determined by Analytical Research. Wood pulp samples are being prepared at the University of Maine using non–sulfur pulping techniques to provide samples for pyrolysis evaluation.

2. **Plans:** Evaluate mainstream smoke from flax and wood models for sulfur–containing components. Conduct pyrolysis evaluations of experimental wood pulps for sulfur–containing products. Conduct subjective evaluations of flax/wood blend papers.

3. **Contributors:** Paper Technology (W. Geiszler), Analytical Research (J. Lyons–Hart, J. Naworal), and Flavor Technology (J. Pflueger).

II. **Objective:** Determine those cigarette paper parameters which most affect cigarette performance and manufacturing processes and set meaningful specifications and tolerances for cigarette papers.

A. **Strategy:** Evaluate the effects of paper properties on cigarette performance attributes (puff count, tar, static burn time, etc.) in order to determine whether tolerances on paper

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specifications are appropriate for Marlboro or other full flavor cigarettes, including determination of paper uniformity requirements for the product.

1. **Results:** An evaluation of the effect of running increased chalk paper on high speed machinery is complete. Standard production 33 Coresta and 46 Coresta cigarette paper containing 30% chalk were run on Protos makers at speeds of 10,000 cigarettes per minute for a period of one month. Machinability and quality data were analyzed and compared to data generated when the same paper types containing 26% chalk were running on the same makers. The results indicate that the 30% chalk paper ran as well at high speeds as the 26% chalk paper. Rates for paper–related defects for the 30% chalk paper were not significantly different than those for the 26% chalk paper. Analytical smoking of Marlboro KS cigarette samples made with the papers confirmed that cigarettes made with 30% chalk paper delivered about 0.5 mg less tar than cigarettes made with 26% chalk paper.

   The relationship between physical paper parameters and tensile strength was investigated. Statistical regression analyses were performed on the set of cigarette papers used in the Response Surface study to determine the parameters that have the largest effect on tensile. For both 33 Coresta and 46 Coresta normal citrate papers, the citrate level, over the specification range for citrate, has a very minute predicted effect on tensile strength. The basis weight has a fairly small predicted effect on tensile over the specification range, but there is a correlation with increasing basis weight resulting in higher tensile strength. The chalk content (or the sheet fiber content) has the greatest effect on tensile strength, and the porosity has the next greatest effect. In both cases, as the chalk content or the porosity increase, the predicted tensile strength of the paper decreases.

   The machinability of papers with extreme tensile properties was investigated by running cigarette papers at varying tensile strengths on a Protos maker at 10,000 cigarettes per minute. Papers with tensile values of about 0.07 kg or higher ran well at the high machine speed; below that level of tensile strength, the paper ran inconsistently or not at all.

2. **Conclusions:** Machinability trials have demonstrated that cigarette paper containing 30% chalk runs effectively at high maker speeds. Papers with tensile strength below 0.07 kg will not run at high maker speeds.

3. **Plans:** Evaluate the effect of chalk content on tar delivery of additional full flavor brands. Determine the variation of key parameters in cigarette paper currently supplied to Philip Morris.

4. **Contributors:** Paper Technology (S. Baldwin, B. Floyd).

B. **Strategy:** Evaluate the effects of paper properties on cigarette performance attributes (puff count, tar, static burn time, etc.) in order to determine whether tolerances on paper specifications are appropriate for low delivery cigarettes.

1. **Results and Conclusions:** Production and delivery analysis of cigarettes made to Marlboro Lights KS and Marlboro Ultra Lights KS specifications are complete.

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Statistical analysis to generate a prediction model is complete. Analysis of data using the prediction model indicate that the effect of chalk on tar delivery of lights and ultra–lights cigarettes is similar to that of full flavor cigarettes, but at a reduced magnitude. Additional statistical analysis of combined cigarette data sets to allow prediction of intermediate tar deliveries is in progress. Production of cigarette models to test the prediction model for light and ultra light cigarettes is in progress.

2. **Plans:** Evaluate the effect of parameter changes on delivery of additional light and ultra light brands. Evaluate the effect of chalk content on tar delivery for additional lights and ultra lights brands.

3. **Contributors:** Paper Technology (S. Baldwin, B. Floyd) and Applied Statistics (M. Ferro, J. Tindall).

C. **Strategy:** Continue studies to evaluate the feasibility of consolidating the seven papers currently being used (excluding Superslims) to two or three papers.

1. **Results and Conclusions:** Mathematical models relating the FTC tar delivery and puff count of full flavor, lights, and ultra lights cigarette designs to four paper parameters — permeability, citrate level, calcium carbonate content, and basis weight — were used to determine specifications for four trial papers. The paper specifications were chosen to provide 0.5 mg FTC tar increments for production cigarettes. The papers will be made in mill trials at Kimberly–Clark during December.

A sampling procedure for monitoring the calcium carbonate content of conventional papers was developed by QA Incoming Materials and Analytical Research. Two grades will be sampled per quarter and analyzed by QA's XRF method. R&D will analyze the same samples by XRF and titration.

2. **Plans:** Evaluate mill trial papers for FTC tar and puff count control on full flavor, lights, and ultra lights product prototypes.

3. **Contributors:** Paper Technology (W. Geiszler) and Analytical Research (K. Torrence).

III. **Objective:** Develop a proprietary cigarette wrapper which will reduce visible sidestream smoke in a full circumference cigarette by 70%, compared to an appropriate control, while maintaining subjective parity.

A. **Strategy:** Optimize the single wrap for Virginia Slims Superslims to achieve an average of 70% sidestream visibility reduction and maintain the current tar delivery target.

1. **Results and Conclusions:** The Superslims brand has been produced with the new 10–064A paper during the past two months. The only changes made to the previous two step 10–062A paper are the slightly higher target level of additive (11 vs. 10.5%) and lower Coresta target, 9.5 Coresta on the 10cm² clamps vs. 10.5 Coresta as measured by the 2cm² clamp. The changes were necessary due to the presence of processing aid in the single step paper (at <0.5%) in order to ensure visibility

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reductions of 70%. Process conditions to be observed by Kimberly–Clark during production of the paper were also referenced in the comment section of the specifications after discussions with K–C. The moisture content of the paper and drying conditions have been found to influence the visibility reduction.

Cigarettes were received through Operations Services from the first week of production with the 10–064A paper. No significant differences were seen, and subsequent samples from QA have shown similar visibility reductions which are achieving the 70% reduction targets. QA smoke data show that the cigarettes give the same deliveries as cigarettes prepared with the two step 10–062A paper.

2. **Plans:** Continue to monitor QA pick–ups of Superslims samples for sidestream visibility reduction.

3. **Contributors:** Paper Technology (B. Goodman, M. Cruise, S. Tafur), Analytical Research (K. Torrence), Tech Services and QE.

B. Strategy: Develop calcium carbonate papers suitable for use with a Virginia Slims Lights type reduced sidestream product.

1. **Results and Conclusions:** A matrix of 47 g/m² low sidestream papers was examined to determine the effect of changing paper parameters on the sidestream reduction and mainstream delivery of 23mm circumference cigarettes. The study was designed to provide the ability to make modifications to the paper used for the Virginia Slims Lights prototypes that were sent out for Consumer testing. A model for predicting extinction coefficients was determined using statistical regression of analyzed paper parameters and measured extinction coefficients of experimental cigarettes. The experimental models gave visibility reductions in the 50 – 67 % range, and the papers had been prepared within the ranges that are feasible for the low sidestream phosphate paper.

   Papers giving higher visibility reductions were also investigated. Reductions of 67 –72 % were achieved with 47 g/m² paper sized with combinations of monopotassium phosphate and acids. The Coresta permeabilities for the sized papers were very low, 1.8 – 2.1 Coresta. Lighter weight paper was sized with the same additives, but burned too slowly to give reliable data. Calendering of monopotassium phosphate coated papers has also been shown to boost sidestream reduction using handmade cigarettes.

2. **Plans:** Coat lower basis weight (42 g/m²) papers with phosphate/acid solutions of low pH to achieve more than 60% visibility reduction on lighter weight papers. Evaluate bobbins of papers which have been calendered.

3. **Contributors:** Paper Technology (B. Goodman, S. Tafur, B. Floyd), Flavor Technology (J. Pflueger), Chemical Research (J. Paine, K. Podraza), Analytical Research (K. Torrence).

C. Strategy: Develop low sidestream papers based on synthetic magnesite and compare these papers to comparable papers containing Baymag magnesite.

1. **Results and Conclusions:** Low sidestream cigarette models made in the Semiworks using three University of Maine machine–made magnesite papers were smoked for head–to–head subjective comparisons. Essentially no differences were detected. The sidestream reductions for the cigarette models prepared with the first two papers exceeded 50%, and those prepared with the more porous paper exhibited reductions of less than 50%. The SBT's for all models were 6.5 to 7.0 minutes.

2. **Plans:** No additional work is planned in this area.

3. **Contributors:** Paper Technology (G. Bokelman, S. Tafur, B. Goodman), Chemical Research (J. Paine, K. Podraza), and Flavor Development (J. Pflueger).

D. **Strategy:** Develop low sidestream papers based on composites containing hydromagnesite and brucite (i.e., the aqueous non–sol–gel process).

1. **Results and Conclusions:** The scale–up effort to synthesize 50 pounds of the hydromagnesite/brucite composition via the non–sol–gel process has been completed on schedule. A total of 64 pounds of the hydromagnesite/brucite composition, synthesized in the Chemical Research Division, was found to be within specifications and was shipped to the University of Maine for papermaking. This material was used to make machine–made paper at the U. of Maine the week of October 19th. Approximately 9,000 feet of a machine–made paper (XPCM) ranging from 45 to 48 g/m², 4 to 5 Coresta, and containing 14 to 16% by weight of the aqueous non–sol–gel material and 14 to 16% by weight Multifex MM calcium carbonate were prepared. A second paper of approximately 5,000 feet (XPCN) ranging from 43 to 45 g/m², 8 to 9 Coresta, and containing 18 to 19% by weight of the aqueous non–sol–gel material and 17 to 18% by weight of Multifex MM calcium carbonate was also prepared. These papers have been slit and studies to determine the burn control agents to be used on them are almost complete.

2. **Plans:** Add burn control agents to both cigarette papers and prepare machine–made cigarettes for evaluation.

3. **Contributors:** Chemical Research (J. Fournier, J. Paine, K. Podraza, J. Seeman), Analytical Research (K. Torrence, L. Thompson, V. Baliga) and Paper Technology (N. Gautam).

E. **Strategy:** Develop low sidestream papers based on amorphous forms of mag carbonates (i.e., the aqueous sol–gel process) using materials which can be scaled–up to produce commercial quantities.

1. **Results and Conclusions:** Process modifications, on a 12–liter scale, have been explored for the scale–up of the aqueous sol–gel process (the reaction of magnesium bicarbonate and magnesium acetate solution with potassium hydroxide at 50°C, optionally followed by heating to 90°C). The use of magnesium hydroxide as the starting material to prepare the magnesium bicarbonate solution resulted in a composition deficient in magnesium hydroxide. The desired hydromagnesite/ magnesium hydroxide ratio is 85/15. A repetition of the procedure incorporating a

calculated increase in the amount of potassium hydroxide has been conducted and the material produced is undergoing extensive analytical evaluation. Hydromagnesite was also investigated as the starting material for the preparation of the magnesium bicarbonate solution. Detailed analyses are pending; however, additional process modifications appear necessary to meet the desired product composition.

2. **Plans:** Optimize 12–liter reaction conditions. Evaluate the product from this process for sidestream reduction, subjectives and ash quality. Prepare approximately 100 pounds of the synthetic hydromagnesite/magnesium hydroxide composition.

3. **Contributors:** Chemical Research (J. Fournier, J. Paine, K. Podraza, J. Seeman), Analytical Research (K. Torrence) and Paper Technology (N. Gautam).

F. Strategy: Develop low sidestream papers based on calcium carbonate with rhombohedral morphology.

1. **Results and Conclusions:** The base papers were treated with equal levels of potassium succinate for direct comparisons of burn rates and sidestream visibility. Mixtures of monopotassium phosphate and succinate or citrate were also applied, and control papers were treated with monopotassium phosphate. Results showed that the puff count increased in one puff increments going from Multifex MM filler to Albacar, Microna 3, and Micro–White 25. The mainstream tar delivery increased correspondingly. Preliminary subjective comparisons have found no major differences among the various calcium carbonates or between papers made at the University of Maine versus those made by Kimberly–Clark. No advantage for sidestream reduction was observed.

2. **Plans:** No further work is planned.

3. **Contributors:** Chemical Research (J. Fournier, J. Paine, K. Podraza, J. Seeman), Analytical Research (K. Torrence) and Paper Technology (N. Gautam, G. Bokelman).

IV. Objective: Conduct studies to assess the feasibility of reducing the levels of those chemical classes in sidestream smoke which are most likely to contribute to irritation.

A. Strategy: Develop analytical methodology in order to determine compositional differences between selected test cigarettes and controls and to relate these differences, if possible, to known irritants or classes of irritants.

1. **Results and Conclusions:** Samples of tobacco flavor extract were analyzed by capillary gc with parallel NPD/FID to determine their characteristic chromatographic patterns and nicotine contents. Also, a gc/ms system was made operational for use in a study on catalytic cracking of possible irritants by the paper filler.

2. **Plans:** Improvements to the headspace gc/ms system and other gc systems for the analysis of sidestream smoke are continuing. Analysis of low molecular weight compounds will be emphasized.

3. **Contributors:** Chemical Research (D. McRae, J. Fournier).

B. **Strategy:** Initiate studies to assess the interactions of selected smoke components with cigarette paper fillers, novel inorganics, and fluxing agents.

1. **Results and Conclusions:** A system utilizing two GCs and a MS has been repaired and reconfigured for use in obtaining detailed information about the interaction of smoke components with cigarette paper fillers, novel inorganics, and fluxing agents. Familiarization with the software and optimization of the system is continuing.

   A trip was made to N.Y. Poly to review their reactor system, in which a heated stream of gas is passed through the material of interest (inorganic filler) and the resulting gaseous products are analyzed using a FT–IR. Acrolein was chosen as the initial smoke component to be evaluated. The influence of inorganic fillers on acrolein will be evaluated at 350°C and 400°C in the reactor.

   Experiments were conducted with MM calcium carbonate and then 50%/50% by weight MM calcium carbonate and two different zeolites of varied pore sizes. Using optimized conditions, i.e., pellet size, reaction temperature, and gas flow rate, the smaller pore size zeolite (~3 A) mixture behaved similarly to the MM calcium carbonate, whereas, the zeolite mixture with a zeolite pore size reported to be slightly larger than the molecular size of acrolein showed a doubling of the acrolein decomposition.

2. **Plans:** The interactions of selected smoke components with cigarette paper fillers, novel inorganics, and fluxing agents will continue to be assessed.

3. **Contributors:** Chemical Research (D. McRae, J. Fournier).

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FILTER
TECHNOLOGY

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PROGRAM NUMBER : 920107
PROGRAM NAME : Filter Technology
PROGRAM COORD. : K. Newman
WRITTEN BY : K. Newman, B. Edwards, D. Laslie, G. Patron, J. Hearn, P. Gauvin, A. Finley
PERIOD COVERED : Fourth Quarter, 1992

Coordinator Summary: Development efforts to provide a domestic alternative to the use of Tela paper in the production of Merit Ultima filters have continued. Paper core concentric filters were made with 100% Terrace Bay cellulose web produced by James River's Gouverneur mill. Analytical results on cigarettes made with these filters compared favorably to the Control. Also, smoking panel results show that this material is subjectively acceptable.

Based on earlier evaluations of Tencel web filters, development efforts have been initiated with DuPont to investigate methods of improving filtration efficiency via increased fiber surface area using their "Sontara" process. Webs are to be manufactured from high pressure hydroentanglement as well as two–stage Tencel fiber/Tencel paper. Other efforts to develop non–woven dry–laid filter webs included the evaluation of hydroentangled webs of cellulose acetate fiber from Courtaulds. The webs in their current configuration showed no filtration efficiency advantage over CA tow.

Surface modifications of paper and other web systems were evaluated. Samples of Tela paper were coated with concentrated extract liquor (CEL) and were converted into paper core concentric filters at American Filtrona for evaluation as an extension of Merit Ultima technology to enhance subjectives. Samples of PM web (75% cellulose acetate/25% cellulose) were laminated with high surface area meltblown polypropylene web at Hollingsworth & Vose (H&V) to increase filtration efficiency. Filters were produced with the modified web which showed 7–10% higher filtration efficiency than Tela paper.

Paper core concentric (PCC) filters with non–wrapped cores from American Filtrona (AFC) were found to achieve the same filtration efficiency as standard paper core concentric filters. Merit Ultima cigarette models were made with the Control and test filters and are being analytically tested. Specifications were established and production was implemented at AFC for a carbon on paper core filter for a 1mg Next product for Japan. Long term physical data shows AFC's process to be capable of maintaining the required filter specifications.

Hardness versus time measurements were replicated for natural–based glycerin type triacetin alternatives. Plasticized filters were made using Estrobond–B, Celanese natural TA, and Unichema natural TA. Subjective analyses done with 8% PZ in the filter showed no differences between the control, Estrobond–B, and the Celanese 100% Natural–based glycerine type triacetin.

Several catalysts to convert CO to CO_2 supplied by Seton Hall are active at room temperature and remain active in the presence of moisture. Dr. Setrak K. Tanilyan of Seton Hall has visited Philip Morris R&D to continue the transfer of synthesis technology from Seton Hall to PM. Plans are to initiate studies at PM in 1) determining flow rate depending of CO oxidation, 2) determining adsorption properties of catalyst for tobacco volatiles, 3) determining CO oxidation rates in the presence of cigarette smoke, 4) determining structural properties affecting catalyst stability.

Celanese has developed a polymer screening technique for testing the selective filtration of a simulated smoke. The technique was tested on polymer coated fiberglass filaments. Differences

were observed during initial screenings. The screening technique was modified to incorporate suggestions made by Philip Morris R&D. Those suggestions increased the throughput of the simulated smoke in the blank control studies.

Efforts continued to characterize PCC filter performance and its analytical/subjective response relationship. King size cigarettes were made with PCC filters and dual Cambridge pad holders were used to collect and separate the core TPM from the peripheral TPM. The pads were submitted to F. Hsu for analysis and comparison.

Product Development Services personnel have prepared and evaluated various packaging materials and designs in response to Engineering and Purchasing requests. Support of new packaging designs have been provided by hand fabrication of prototypes for: unique packaging concepts and designs, Booklet Pack, Red Express, Recloseable Soft Pack, B&H Deluxe hinge lid box in a 1x10 cigarette packing configuration, and a 2x5 configuration hinge lid box for Marlboro (Korea).

An all aluminum pack design has continued to be a major effort during this quarter. A shift in emphasis has been made from drawn to folded box designs. Silk screen printed laser cut blanks have been prepared and tooling is being developed to form the box.

I. **Objective:** Develop web cigarette filtration system(s) which offer the consumer perceived benefits when incorporated into new cigarette systems.

A. **Strategy:** Develop a non–woven wet–laid sheet of cellulose acetate and cellulosic fibers.

1. **Results:** PM web dual filters were evaluated on Merit 3mg products and were found to be subjectively inferior to CA tow. Additional application work is underway on Lark Ultra Lights using web inner filters in a plug–space–plug design.

   A series of filter webs were produced at the University of Maine in October. These experimental webs included 75%/25% Cellulose Acetate/Softwood, 100% cotton, 100% eucalyptus, and 100% softwood web. The use of different fibers in the filter webs is directed to improve the filter efficiency and the subjective response over the current web materials. These webs are being evaluated in–house for machinability and filtration properties.

2. **Plans:** Produce pilot quantities of web materials at the University of Maine incorporating CA fibrids from Courtaulds.

3. **Contributors:** N. Gautam, D. Laslie, P. Gauvin, K. Newman

B. **Strategy:** Develop a domestic source of 100% wood pulp filter web which qualifies as an alternate material to Tela paper in the production of Merit Ultima filters.

1. **Results:** Paper core concentric filters were made with 100% Terrace Bay cellulose web produced by James River's Gouverneur mill. Analytical results on cigarettes made with these filters compared favorably to the Control. Also, smoking panel results show that this material is subjectively acceptable.

2. **Plans:** Write completion report including process and material specifications for future reference.

3. **Conclusions:** Acceptable domestic sources of Tela paper are available if cost, quality, or source security issues arise with the current Ultima material.

4. **Contributors:** N. Gautam, D. Laslie, K. Newman, J. Pflueger

C. **Strategy:** Develop a non–woven dry–laid filter web of Tencel solvent–spun cellulose fibers.

1. **Results:** Based on earlier evaluations of Tencel web filters, development efforts have been initiated with DuPont to investigate methods of improving filtration efficiency via increased fiber surface area using their "Sontara" process. Webs are to be manufactured from high pressure hydroentanglement as well as two–stage Tencel fiber/Tencel paper. A joint confidentiality agreement between PM and DuPont has been completed.

   An additional 1,000 yards of 1 oz. per square yard 100% Tencel web has been obtained from DuPont. Discussions are in progress within Filter Technology to determine which filter production method should be evaluated with this material.

   A joint development meeting on the production and use of Tencel webs for the manufacture of new cigarette filters was held with the DuPont Fibers division and Courtaulds. A development plan was prepared which involves the manufacture of nine trial Tencel webs by DuPont. Work is in progress to implement this plan.

   Hydroentangled webs of cellulose acetate fiber from Courtaulds were evaluated and showed no filtration efficiency advantage over CA tow in their current configuration.

2. **Plans:** Evaluate filtration characteristics of hydroentangled dry laid webs to be provided by DuPont. Investigate wet laid fibrillated Tencel web technology as an alternate method.

   Produce an improved cigarette model with a 100% Tencel filter. Conduct trials with the current 100% Tencel web to determine if alternate forming methods will yield improved Tencel filters. Finalize the development plan on the production of new Tencel webs between DuPont, Courtaulds, and PM.

3. **Conclusions:** Tencel web can be used to produce filters with acceptable end appearance and filter efficiencies which are better than standard cellulose acetate filters. DuPont has the capability to produce a number of alternate Tencel webs which should allow PM to produce Tencel filters which show further improvements in filtration efficiency.

4. **Contributors:** B. Edwards, P. Gauvin, D. Laslie, K. Newman

D. **Strategy:** Develop paper and cellulose acetate/cellulose web systems with surface modifications to alter sensory and analytical performance.

1. **Results:** Samples of Tela paper were coated with concentrated extract liquor (CEL) and were converted into paper core concentric filters at American Filtrona for evaluation as an extension of Merit Ultima technology to enhance subjectives. Due

to limited sample quantities, only hand–made cigarettes were evaluated. Additional CEL on Tela web samples are to be coated on the R&D RL process for a more thorough prototype analysis.

Samples of PM web (75% cellulose acetate/25% cellulose) were laminated with high surface area meltblown polypropylene web at Hollingsworth & Vose (H&V) to increase filtration efficiency. Filters were produced with the modified web which showed 7–10% higher filtration efficiency than Tela paper. The H&V laminating process will require additional development.

2. **Plans:** Characterize and analyze CEL paper core concentric cigarette models to identify and exploit potential benefits.

   Meet with H&V to discuss their capabilities and proposals for developing an improved web filter material.

3. **Contributors:** D. Laslie, B. Edwards, P. Gauvin, K. Newman, G. Patron

E. **Strategy:** Develop manufacturing processes and operations to produce filters from new filter media.

1. **Results:** Bobbins of PM web and Tela paper were sent to Baumgartner, Mebane, N.C. to make plug–space–plug (web+carbon+CA) filters for consumer testing on Lark Ultra Lights prototypes.

   Paper core concentric filters with non–wrapped cores from American Filtrona (AFC) were found to achieve the same filtration efficiency as standard paper core concentric filters. Merit Ultima cigarette models were made with the Control and test filters and are being analytically tested.

   Specifications were established and production was implemented at AFC for a carbon on paper core filter for a 1mg Next product for Japan. Long term physical data shows AFC's process to be capable of maintaining the required filter specifications.

2. **Plans:** Qualify Baumgartner as a commercial supplier of plug–space–plug filters. Evaluate PSP filter quality, acceptability and fitness for use on Lark Ultra Lights.

   Analytically and subjectively compare paper core concentric filters with non–wrapped cores versus standard paper core concentric filters on Merit Ultima. Determine any cost/process/product benefits.

3. **Contributors:** D. Laslie, K. Newman

II. **Objective:** Investigate acceptable alternate cellulose acetate (CA) plasticizers to replace triacetin (TA). Investigate natural–based glycerin triacetin as a replacement for synthetic–based glycerin triacetin.

A. **Strategy:** Investigate natural–based glycerine type triacetin as an alternative to the triacetin currently used.

1. **Results:** Hardness versus time measurements were replicated using Estrobond–B, Celanese natural TA, and Unichema natural TA plasticized filters. The test filters

were made to the same plug–maker machine speeds. The dry filter weights were held constant for a given tow item at a given RTD. Filters were made using FT–666, FT–107, and FT–555 tow at 8% and 6% PZ levels. Again, no significant difference was noted for the various plasticizers at 8% or 6% PZ levels, but there were measurable differences in hardness when comparing 8% levels to 6% levels. The trend suggesting different hardness per unit of plasticizer, with Estrobond–B showing the largest response and Unichema showing the smallest response, was not seen in the second set of data. The Unichema product was marginally different.

2. **Plans:** Flavor Development will have two Marlboro cigarette models made for subjective analyses. The models will have 8% PZ in the filters. The control will use Estrobond–B and the test will be the Celanese 100% natural glycerine product. A POL will follow, if the results are positive.

3. **Conclusions:** Subjective analyses done with 8% PZ in the filter showed no differences between the control, Estrobond–B, and the Celanese 100% Natural–based glycerine type triacetin.

4. **Contributors:** K. Deane, A. Finley, R. Hale, K. Lam, J. Ruziak

III. **Objective:** Evaluate the feasibility of developing a catalyst to convert CO to CO_2 in a cigarette system.

A. **Strategy:** Test CO removal catalysts supplied by Seton Hall University.

1. **Results:** Several catalysts supplied by Seton Hall are active at room temperature and remain active in the presence of moisture. These catalysts are lower in cost compared to noble metal catalysts. The activity of the catalysts can be increased under adiabatic conditions, with increased CO flow rates, or by increasing the initial temperature. After aging for 110 days, the $MnCo(O)_x$ catalyst converted approximately 30% of the CO flow to CO_2 under adiabatic conditions. Dr. Setrak K. Tanilyan of Seton Hall has visited Philip Morris R&D to continue the transfer of synthesis technology from Seton Hall to PM.

2. **Plans:** Continue the transfer of synthesis technology from Seton Hall to PM.

Initiate studies at PM in 1) determining flow rate depending of CO oxidation, 2) determining adsorption properties of catalyst for tobacco volatiles, 3) determining CO oxidation rates in the presence of cigarette smoke, 4) determining structural properties affecting catalyst stability.

The appropriate filter development criteria will be maintained on an on–going basis.

3. **Contributors:** A. Finley, D. Kellogg, K. Koller, K. Podraza, J. Seeman, K. Shafer, J. Wooten

IV. **Objective:** Design evaluate, and develop new filter concepts that provide improved subjective benefits to our consumers.

A. **Strategy:** Develop fibers with new/unique filtration properties.

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1. **Results:** Celanese has developed a polymer screening technique for testing the selective filtration of a simulated smoke. The technique was tested on polymer coated fiberglass filaments (used only in screening experiments). The simulated smoke contains 16 components with concentrations similar to ratios in tobacco smoke. The technique measures the amount of each smoke component allowed through the filter simulation chamber. Differences were observed during initial screenings. The screening technique was modified to incorporate suggestions made by Philip Morris R&D. Those suggestions increased the throughput of the simulated smoke in the blank control studies.

   A meeting was held with Courtaulds to discuss the possibility of production of trial quantities of a Tencel tow suitable for cigarette filter production. As a result of this meeting, Courtaulds will be conducting a detailed evaluation of Tencel tow production. They have agreed to provide PM with the results of this evaluation which will cover type of tow that can be made as well as expected development and production costs.

2. **Plans:** Maintain close communications with research personnel at Celanese. Meet in early December for update on polymer screening.

   Receive and evaluate Tencel tow proposal from Courtaulds.

3. **Contributors:** A. Finley, P. Gauvin, K. Newman, J. Hearn

B. **Strategy:** Develop an understanding of and the ability to model the delivery of smoke components from cigarette filter with paper core concentric (PCC) filters.

1. **Results:** King size cigarettes were made with PCC filters and dual Cambridge pad holders were used to collect and separate the core TPM from the peripheral TPM. The pads were submitted to F. Hsu for analysis and comparison.

2. **Plans:** Evaluate written response from F. Hsu that summarizes the results of the GC analyses of the core and peripheral TPM. Evaluate the data to determine if partitioning of smoke components occurs in PCC filters. Investigate to determine if analytical data is consistent with subjective observations. Proceed with calculations of core/periphery deliveries to determine the extent of current predictive capabilities.

C. **Strategy:** Investigate utilizing uniformly pre–perforated mouthpiece paper with pre–perforated tipping paper on Parliament products to eliminate on–line laser perforation.

1. **Results:** A bobbin of Parliament mouthpiece papers was perforated by micro–laser and inner components for Parliament 100's were manufactured in Semiworks. The appearance of the combined filter rods was unacceptable. The laser perforations in the Parliament mouthpiece paper were visible through the tipping and presented an aesthetic concern.

2. **Plans:** Investigate other means of obtaining uniform perforation patterns or high porosity in specific regions or bands in high basis weight papers such as Parliament mouthpiece paper.

3. **Contributors:** J. Ryder, J. Hearn

V. **Objective:** Provide innovative packaging designs, materials, and manual fabrication skills to support new product introductions, strategic goals as related to environmental issues, and existing brands improvement.

A. **Strategy:** Develop packaging that more easily degrades after use than the current packaging. Conceivably, this could be achieved by developing new adhesive systems for paper fiber and/or fiber formation into packages.

1. **Results:** Moulded Fiber Technology of Westbrook, MA has not responded to requests for materials and information. Therefore, further activities with them will be curtailed and alternative vendors will be pursued. Another vendor was identified during attendance at Pack Expo '92 and material samples were requested.

   Information has been requested from Poly–Bond, Inc. to determine their capabilities in the technology of adhesive application to webs and films.

2. **Plans:** Continue efforts to identify a vendor who has a process for making a molded pack from paper fibers.

   Continue evaluating available technologies and packaging materials for applicability to "biodegradability."

B. **Strategy:** Manually fabricate innovative packaging designs to support the development of new brand and improve existing brands.

1. **Results:** Product Development Services personnel have prepared and evaluated various packaging materials and designs in response to Engineering and Purchasing requests. Support of new packaging designs have been provided by hand fabrication of prototypes for: unique packaging concepts and designs, Booklet Pack, Red Express, Recloseable Soft Pack, B&H Deluxe hinge lid box in a 1x10 cigarette packing configuration, and a 2x5 configuration hinge lid box for Marlboro (Korea).

   An all aluminum pack design has continued to be a major effort during this quarter. A shift in emphasis has been made from drawn to folded box designs. Silk screen printed laser cut blanks have been prepared and tooling is being developed to form the box.

2. **Plans:** Continue to provide support in the form of mock–up fabrication, conceptualization, and participation on the Packaging Innovation Team. Provide packaging mock–ups to PED for Consumer Research testing in early December. Continue to monitor innovative packaging materials/concepts available in the literature and through trade shows and other professional contacts.

3. **Contributors:** J. Hearn, R. Newsome